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COTTON WASH-WEAR RESEARCH

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Compiled by
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WASH-WEAR COTTONS STILL IN DEVELOPMENT STAGE DESPITE PHENOMENAL SUCCESS

The wash-wear and minimum-care treatments which have become so popular since 1955 are unquestionably the biggest thing to happen to cotton products since the advent of sanforization some thirty years ago. Good as are many of the wash-wear cottons now on the market, one result of their popularity is to demonstrate conclusively the need for still better wash-wear cotton fabrics and wash-wear garments.

For this reason there is probably more research effort devoted at the present time to the improvement of wash-wear treatments than to any other single area of cotton utilization research. Independent, governmental, and industrial research agencies both in this country and abroad are working on the various problems.

Scientists at the Southern Utilization Research and Development Division, foreseeing the potential value of wash-wear or wrinkle-resistant cotton fabrics, have been interested in this area of investigation since the establishment of the Southern Regional Laboratory in 1939. Early in the decade of the 1950's, when the great need and opportunities for wash-wear cottons became more apparent, Southern Division research was broadened and intensified.

The key phenomenon in the production of wash-wear and minimum-care cottons is crosslinkage, the formation of bonds or links tying together two or more of the long, streamlined, molecules which give the cotton fiber its special characteristics. Theoretically, the formation of such bonds, or crosslinks, holds the molecules in position, thus rendering the fibers less susceptible to distortion, or creasing. Crosslinking is fundamental to the production of wrinkle-resistant cotton fabrics, as well as a number of other chemical modifications to impart new and desirable properties to cotton for other specific uses.

In spite of the usefulness of crosslinkage, however, this technique was applied to cotton only on a small scale prior to 1955. This delay was not because of the lack of need, but rather from a dearth of the scientific information necessary to apply the process successfully. In other words, we had jet planes, atom bombs, and other modern developments before we could successfully manufacture practical and useful crosslinked cotton products. Even today, our best methods for crosslinking cotton have serious deficiencies.

A great deal of fundamental knowledge of the physical structure, the chemical composition, and the behavior of the cotton fiber has been developed at the Southern Division, much of it basic to the development of wash-wear treatments and other types of chemical modification. This information has been made available to researchers outside the Division through technical publications.

Among more recent publications on the fundamental aspects of crosslinkage in wash-wear treatments is one in which Division scientists report that the effectiveness of the crosslinking treatments depends more on the number of the crosslinks than on their location. Stability of the bonds formed influences the durability of the treatment.

Since publication of the first paper from the Southern Division on wash-wear research, 44 additional scientific papers on this subject have been published in technical journals by Division researchers, reporting explorations in all areas of such investigations. Some of the findings have already found application in commercial operations, and others are being studied with a view to adoption by industry. We now have 15 other papers either accepted for publication or in preparation, and a number of research projects underway which can be expected to develop further information.

One serious problem threatening the acceptability of some fabrics with wash-wear finishes was their tendency to pick up soil during laundering. Research at the Southern Division showed that certain acrylic and silicone polymers used in finishing softened in the presence of heat, and picked up soil which could not be removed afterward. Since then industry has generally adopted polyethylene instead, as recommended by Southern Division scientists and covered by a USDA patent.

In the course of investigations at the Southern Division many compounds have been studied for their applicability as crosslinking agents to impart wash-wear properties to cotton, and the findings published. Some of these compounds offer attractive advantages, and it is understood that some of them are now in commercial use.

Listed here among the papers already accepted by technical journals and awaiting publication is one on "Formaldehyde Treatment of Partially Swollen Cotton," which describes a new Form-D process whereby the treated fabric has good dry (as well as wet) wrinkle recovery. The Form-W process using formaldehyde, previously announced, has excellent wet wrinkle recovery, but very little resistance to wrinkling while in the dry state.

Investigation of the loss-of-strength characteristics of wash-wear finished fabrics has led to the publication of several papers. One of these reports that mercerization after curing of the treated fabric minimizes loss of both tear strength and abrasion resistance in the case of many of the more common finishing agents. Also of great interest is the report, now awaiting publication, on the application of low tension to the fabric during processing and curing to reduce strength losses. These studies also showed that selection of high-strength, high-elongation cottons, and premercerization increased the toughness.

While investigations of other phases of wash-wear go on, the search for new and better finishes has not been neglected. One of the most promising of these is tris(1-aziridinyl) phosphine oxide (APO). The finish imparts a high degree of wrinkle-resistance, and is unusually stable to laundering. A disadvantage is that white goods treated with APO show a tendency toward yellowing when ironed after a chlorine bleach. Its other properties are so good, however, that several chemical firms in this country are considering the commercial production of APO.

This booklet contains abstracts summarizing papers published or soon to be published on these and other research developments of the Southern Division in the wash-wear field. We feel that they are particularly valuable, especially the findings on fundamental principles.

The phenomenal public acceptance of wash-wear and minium-care cottons now on the market is in itself a clear indication of the need for further improvement in these products. P.J. Fynn, Research Director of J. C. Penney Co., has defined the goal of wash-wear in this manner: "Wash-and-wear, in my opinion, can have only one meaning. It applies only to a garment which, when laundered according to a reasonable procedure without any ironing, would be worn willingly by a reasonably fastidious person, in public, for activities appropriate to the garment." There is a great deal still to be learned before this standard can be generally met in commercial production.

In the meantime, however, wash-wear developments have already had a pronounced impact on consumption of cotton. The National Cotton Council estimates that in 1960 wash-wear was responsible for the consumption of a million bales which would not have been used without these developments. Cotton has shown an upward trend in most areas from 1956 to the present. Increases have been greatest in apparel uses where the development of wash-wear cotton fabrics could be expected to have the most effect. In 1947, apparel accounted for 36% of total cotton consumption; apparel accounted for more than 52% of domestic cotton consumption in 1959. In some types of apparel, such as women's street dresses, men's dress shirts, and children's and infants' dresses, wash-wear finishes are credited with helping to prevent cotton's losses of markets. Measured in volume of bales consumed cotton has shown distinct advances, while percentages of the total have remained approximately the same or advanced slightly. In some other types of apparel cotton has made some noteworthy advances. Between 1947 and 1958, the cotton's percentages of the total have risen from women's skirts from 7% in 1947 to 46% in 1958; women's blouses, from 14% in 1947 to 72% in 1958, and men's sport shirts, from 43% in 1947 to 75% in 1958.

It is notable that about 81% of the current cotton consumption is in uses where individual consumers have a voice in fiber selection, and in uses which are responsive to quality improvements and sales promotion. This may be taken as an indication that further improvements can be expected to bring an increased demand for a fiber which is already a favorite because of its many good qualities.

PREFACE

An abstract bibliography of all publications reporting research by the Southern Division on the utilization of cotton is now being prepared. For the convenience of those who may be interested in a specific area of work, however, we have also prepared listings of publications on research in some special fields.

We have assembled in this booklet abstracts of our publications and patents on wash-wear, and on crosslinking and other phenomena basic to wrinkle resistance and other wash-wear properties. We are also including manuscripts pertaining to this area of research, as well as patent applications now pending. Patents have been assigned by the grantees to the Secretary of Agriculture for licensing to citizens of the United States on a royalty-free basis.

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PUBLICATIONS PLANNED

A FURTHER STUDY OF HYPOCHLORITE RESISTANT MELAMINE TYPE FINISHES

Vail, Sidney L.; Frick, J. G., Jr.; and Reid, J. David

Previous work in this field has been expanded to include additional substituted amino-s-triazines. A more thorough understanding of the mechanism of yellowing of melamines and melamine finishes by hypochlorite treatment has been sought. Results obtained from chlorination of melamine finishes on fabric have been compared to results obtained from chlorination of melamines in vitro. Data of a practical nature concerning the use of N, N', N" - trimethylmelamine-formaldehyde products in the finishing of cotton textiles is also presented.

ACID RESISTANCE OF WASH-WEAR FINISHES FOR COTTON

Arceneaux, R. L.; Andrews, B. A. K.; Frick, J. G., Jr.; and Reid, J. David

The acid or sour resistance of several nitrogenous finishing agents and the finishes they produce on cotton have been investigated. A correlation between the structure of the agent and the acid resistance of the finish is shown.

STABILITY OF THE URON RING IN WASH-WEAR FINISHES

Arceneaux, R. L.,; and Reid, J. David

The use of uron derivatives as wrinkle-resistant finishes for cotton was investigated. Although the uron ring in 1,3-dimethyluron is hydrolized by acid catalyst the resultant intermediate forms only a single link with cellulose. Application of pure bis-methoxymethyluron to cotton fabrics with acid catalyst produces a wash-wear finish with good durability to laundering and resistance to chlorine damage. Use of an unpurified uron mixture produces a finish with good durability but poor resistance to chlorine damage.

FLUORESCENT WHITENING AGENTS IN THE APPLICATION OF WASH-WEAR FINISHES TO COTTON

Reinhardt, Robert M.; Fenner, Terrence W.; Reid, J. David; and Furry, M. S.

Cotton which has been crosslinked to impart wash-wear properties is resistant to whitening by fluorescent agents. To achieve the whitening effect with these agents, the cotton must be "dyed" with the agent prior to crosslinkage or the agent may be applied with the crosslinking agent.

in the same pad bath. Three different methods of applying whitening agents during the finishing of cotton to impart wash-wear properties were investigated. Changes in the properties of the fabric and durability of the whitening effect were determined. (A cooperative investigation with the Institute of Home Economics.)

IMPARTING DRY WRINKLE RESISTANCE TO COTTON TREATED IN THE SWOLLEN STATE WITH FORMALDEHYDE

Fenner, Terrence W.; Reinhardt, Robert M.; and Reid, J. David

A process has been studied for imparting dry wrinkle resistance to Form W cotton, that is, cotton which has been treated with formaldehyde in the swollen state. By this process, Form W cotton is impregnated with an acid or latent acid catalyst, subjected to a heat treatment and afterwashed. Many variables in the treatment that affect the final product were examined. As compared with Form W cotton, the treated Form W cotton has greatly improved dry wrinkle resistance and thus a higher wash-wear rating after being tumble dried. Breaking strength and tearing strength were somewhat decreased, but wet wrinkle resistance, moisture regain and durability of the finish were practically unaffected.

THE USE OF COREACTANT CURING AGENTS WITH A DIEPOXIDE TO IMPART WRINKLE RESISTANCE TO COTTON FABRICS.

Reinhardt, Robert M.; Kullman, Russell M. H.; Moore, Harry B.; and Reid, J. David

Although the use of coreactant curing agents with epoxides is common in the production of plastics, little has been published on the use of these agents with epoxides to impart wrinkle resistance to cotton fabrics. Such materials coreact to become a part of the finished product and introduce linkages and chemical groups which differ from those obtained when a true catalyst is used. Several coreactants including phthalic anhydride, citric acid, 1-aminoethyl-2-methyl-imidazoline, triethanolamine, and potassium thiocyanate, were investigated, using 1,3-diglycidylglycerol as the diepoxide. In some cases mere deposition seemed to occur while in other cases crosslinkage was accomplished. Some indications of the types of reaction were obtained from infrared data and from the responses of the treated fabrics to various classes of dyes. In some cases coreaction between the diepoxide and the curing agent appeared to take place at relatively low temperatures producing improved wrinkle resistance. Phthalic anhydride was used to investigate this aspect in more detail. For comparison with the coreactant materials, several acid salts were used as catalysts, and the results are reported. Among the more suitable acid catalysts are magnesium perchlorate, aluminum sulfate, and zinc fluoborate.

The usual textile tests were made. The inorganic acid catalysts produced fabrics with excellent durability and wrinkle resistance. Tearing strength losses, however, were greater with epoxide-treated cotton

than those observed, at equivalent crease recovery angle, when typical methylolamide finishing agents with catalyst were used. The durability to alkaline solutions of some of the epoxide finishes applied with coreactant curing agents was not as good as that of fabrics prepared with the inorganic acid catalysts. As would be expected, the tearing strength and wrinkle resistance of epoxide-treated cotton can be improved by inclusion of additives, such as polyethylene, acrylic polymer or silicone in the finishing bath.

CROSSLINKING ETHERIFIED COTTONS WITH DIMETHYLOL ETHYLENEUREA

Kullman, Russell M. H.; Frick, John G., Jr.; Reinhardt, Robert M.; and Reid, J. David

The effects of crosslinking with dimethylol ethyleneurea on several etherified cotton fabrics--carboxymethylated, hydroxyethylated, methylated, and cyanoethylated cottons--were studied and compared with unmodified cotton. The effect of replacing cellulosic hydroxyl groups on the reactivity toward dimethylol ethyleneurea is dependent on the nature of the substituent and the swellability of the etherified fiber. Hydroxyethylated and methylated cottons exhibit greater crosslinking efficiency with dimethylol ethyleneurea than does unmodified cotton. High moisture regain in conjunction with high wet and conditioned wrinkle recovery is possible with hydroxyethylated cotton. Increased extensibility observed in etherified cottons is directly related to the alkali concentration used in the etherifying reaction. The rate of strength loss on crosslinking with unit improvement in crease recovery is greater for etherified cottons, except hydroxyethylated, than for unmodified cotton.

CONTROL OF FORMALDEHYDE RELEASE FROM FABRIC FINISHED WITH DIMETHYLOL ETHYLENEUREA

Reid, J. David; Reinhardt, Robert M.; Fenner, Terrance W.; and Harris, James A.

Several methods for diminishing the amount of formaldehyde released from fabric treated with dimethylol ethyleneurea (DMEU) were studied. Afterwashes, aftertreatments, and finishing pad bath additives were investigated. The formaldehyde evolved was measured after an accelerated storage test. The effect of treatments to control formaldehyde release on wrinkle resistance and chlorine resistance of the wash-and-wear fabric was determined. The study was limited to cotton cloth treated with DMEU and zinc nitrate catalyst, but the results are believed to be generally applicable to fabric finished with the various types of N-methylol finishing agents. Oxidative and alkaline afterwashes were effective in controlling formaldehyde evolution with no adverse effect upon wrinkle resistance or chlorine resistance. Many of the other treatments studied were inefficient in satisfactorily diminishing the release of formaldehyde or produced undesirable side effects on important wash-wear properties of the fabric. Dicyandiamide, ethyleneurea, urea, and sodium metabisulfite as aftertreatments all reduced formaldehyde release in the finished cotton.

FURTHER AUTOMATION OF THE SRRL WRINKLE RECOVERY TESTER

Haydel, Chester H. and Sloan, Julia M.

With the addition of the pressing feature and the 3-band program timer, the Southern Regional Research Laboratory tester for wrinkle recovery by the vertical strip method becomes fully automatic except for sample insertion and folding. Its use should result in improvements in overall efficiency and economy in large scale testing of this property, particularly with respect to wash-wear developments. Not only does it make possible greater productivity by a single technician, but the results obtained could be more objective and comparable between different technicians and laboratories.

THE IN SITU POLYMERIZATION OF ETHYLENE UREAS AND ETHYLENE AMIDES WITHIN THE FIBERS OF COTTON

Jones, F. B.; Hammon, H. G.; Leininger, R. I.; and Heiligman, R. G.
Contractor: Battelle Memorial Institute, Columbus, Ohio.

A research program has been carried out to investigate a new approach to cotton finishing. This approach has involved a study of various diaziridinyl compounds, such as those obtained by reacting ethylene imine with organic diisocyanates, diacid chlorides, and bischloroformates as potential crosslinking agents for cotton cellulose to produce "minimum care" or "wash-wear" fabric finishes. Over 25 different aziridinyl compounds were prepared experimentally and screened for utility on the basis of water solubility and effect on such fabric properties as crease-recovery angle, tear strength, breaking strength and elongation, flex abrasion, wash fastness, and chlorine retention. The experimental finishes were applied to an 80 x 80-count cotton print cloth, using laboratory-size padding and drying equipment.

The diaziridinyl compounds based on aliphatic diisocyanates and bischloroformates were found to demonstrate the greatest utility for the contemplated application, particularly those compounds prepared from hexamethylene-1,6-diisocyanate and mono- and diethylene glycol bischloroformate precursors. Increases in crease-recovery angle in the range of 30 to 60 percent were achieved with these finishes under conditions of application generally similar to those employed for commercial polymethylol type finishes.

While these particular diaziridinyl finishes impart considerable crease resistance to cotton cloth, they were found to generally cause less impairment of other fabric properties than an experimentally applied commercial polymethylol cotton finish (CEU resin). The diaziridinyl finishes, being nitrogen-containing compounds, demonstrate a strong tendency to retain chlorine after chlorine bleaching. Further development of curing techniques for application of these finishes should be considered as a possible means of reducing chlorine retention.

ARS 72-18, INVESTIGATION OF THE EFFECT OF PREPARATORY FINISHING PROCESSES AND RESIN TREATMENT ON THE TEAR STRENGTH OF VARIOUS COMMERCIAL COTTON FABRICS

ARS 72-19, INVESTIGATION OF MODIFICATIONS OF YARN AND FABRIC STRUCTURE REQUIRED FOR THE IMPROVEMENT OF TEAR STRENGTH CHARACTERISTICS OF COTTON FABRICS

ARS 72-20, EVALUATION OF THE EFFECTS OF COTTON FABRIC STRUCTURE AND RESIN TREATMENT ON THE TEAR STRENGTH OF VARIOUS COMMERCIAL COTTON FABRICS

These three publications present in their entirety these three phase reports previously published in condensed form as:

2012. FABRIC STRUCTURE AND WASH-AND-WEAR
/Influence of Fabric Structure on Tear Strength of Resin-Treated Cotton Fabrics/
Stavrakas, E. James; and Platt, Milton M.
Contractor: Fabric Research Laboratories, Inc., Dedham, Mass.
Textile Inds. 124, No. 10, 141-160. 1960
(Which see for an abstract.)

ACCEPTED FOR PUBLICATION

DIMETHYLOLTRIAZONE FINISHING AGENTS WITH LONG-CHAIN ALKYL SUBSTITUENTS FOR COTTON TEXTILES

Vail, Sidney L.; Frick, J. G., Jr.; Murphy, Paul J., Jr.; and Reid, J. David
Am. Dyestuff Reptr. (In Press)

Three new triazones, with n-octyl, n-dodecyl, and n-octadecyl groups as 5-substituents, have been prepared. Dimethylol derivatives of these triazones were examined as reactive finishing agents for cotton fabrics. Of these, only the one derived from octyltriazone produced wrinkle resistance. The degree of wrinkle resistance was low and it is concluded that this molecule is too large for more than partial access to the fiber and can, therefore, produce only limited wrinkle resistance. The dimethylol derivatives of the dodecyl-and octadecyltriazone were too large to give wrinkle resistance but did react to give a softening effect to the fabric. The octadecyltriazone produced only a small amount of water repellency whereas the other agents produced less or none at all.

DIHYDROXYETHYLENEUREAS OF WRINKLE-RESISTANCE FINISHING AGENTS FOR COTTON

Vail, Sidney L.; Murphy, Paul J., Jr.; Frick, J. G., Jr.; and Reid, J. David
Am. Dyestuff Reptr. (In Press)

The ring hydroxyl groups of 4,5 dihydroxy-2-imidazolidinones (dihydroxyethyleneureas) have been shown to be chemically active and to produce crosslinks similar to these produced by conventional N-methylol crosslinking agents. The properties of fabrics treated with some dihydroxyethyleneureas have been studied and compared to fabrics treated with dimethylolethyleneurea. 1,3-Dimethyl-4,5-dihydroxy-2-imidazolidinone, believed to be a new compound, has been prepared and found to produce wrinkle-resistant cotton fabrics. These fabrics were highly resistant to chlorine damage and had good resistance to acid souring. Wrinkle-resistant fabrics were also produced from 4,5-dihydroxy-2-imidazolidinone. These fabrics, however, were yellowed during curing and were highly susceptible to chlorine damage.

FORMAMIDE-FORMALDEHYDE PRODUCTS AS WRINKLE-RESISTANCE FINISHING AGENTS FOR COTTON FABRICS

Vail, Sidney L.; Frick, J. G., Jr.; Murphy, Paul J., Jr.; and Reid, J. David
Am. Dyestuff Reptr. (In Press)

Formamide-formaldehyde finishes have been used previously to some extent to impart wrinkle and shrinkage resistance, and to increase the wet strength of regenerated cellulosic fabrics. Little has been published, however, concerning the properties of cotton treated with such finishing agents. The properties of cotton fabric treated with these

agents are described and it is concluded that the finish produces wash-and-wear fabrics, but the finished fabrics are susceptible to damage from hypochlorite bleaching. For a methylolamide type of finish, it shows a very high resistance to both acidic and basic hydrolysis. The mechanism of wrinkle resistance is believed to be due to crosslinks of both formaldehyde and formamide-formaldehyde types. Crosslinks produced by two methylol groups attached to the same amido nitrogen atom are unusual.

A STUDY OF HYPOCHLORITE RESISTANT MELAMINE TYPE FINISHES
Vail, Sidney L.; Frick, J. G., Jr.; and Reid, J. David
Am. Dyestuff Reptr. (In Press)

The commercial wash-wear and wrinkle-resistance finishing agents for cotton fabrics based on melamine-formaldehyde products are widely used but many suffer the disadvantage that the treated fabrics yellow noticeably when washed using hypochlorite bleach. This problem has been studied and the cause of yellowing of fabrics treated with melamine type agents has been attributed to the reaction of hypochlorite with primary amino groups in the finished fabric. Exclusion of these groups and elimination of the yellowing was accomplished by placing stable substituents, methyl or 2-hydroxyethyl groups, on each amino nitrogen of the s-triazine ring before reaction with formaldehyde. The properties of the fabrics treated with the formaldehyde reaction product of acetoguanamine, N,N-bis(2-hydroxyethyl) melamine, N,N'-dimethyl-N"-bis (2-hydroxyethyl) melamine and N,N',N"-trimethylmelamine are presented and compared with those of fabrics treated with some commercial methylolmelamines and methylated methylolmelamines. The preparation of these melamines and some of their properties are also described.

STUDIES OF WRINKLE RESISTANCE FINISHES FOR COTTON TEXTILES: III. AN ACCELERATED STORAGE TEST FOR WASH-AND-WEAR COTTON FABRICS
Arceneaux, Richard L.; Gautreaux, Gloria A.; Reinhardt, Robert M.; and Reid, J. David
Am. Dyestuff Reptr. (In Press)

An accelerated test procedure to determine the effect of storage on wash-wear cotton fabrics was developed. Fabrics finished with five N-methylol finishing agents, each with three different catalysts, were stored for 16 hours in sealed jars at 60°C. and 100% relative humidity. The effects produced were comparable to those of prolonged normal storage. Optimum test conditions were established by determining the effect of storage time and temperature on fabrics finished with dimethylol ethyleneurea and zinc nitrate catalyst.

WRINKLE RESISTANT PROPERTIES OF DIALDEHYDE COTTON
Mack, Charles H.; and Reeves, Wilson A.
Textile Research J. (In Press)

The effect of periodic acid oxidation on the wrinkle resistance of cotton fabric has been investigated. In the range of oxidation where basic fabric properties are preserved the dry wrinkle recovery angle

increases with increasing oxidation. The change in wet wrinkle recovery angle for these fabrics is negligible, suggesting that crosslinks which are evident in the dry state are broken in the presence of water. An explanation involving hemiacetal and hemialdal formation is advanced.

FORMALDEHYDE TREATMENT OF PARTIALLY SWOLLEN COTTON

Chance, Leon H.; Perkins, Rita M.; and Reeves, Wilson A.
Textile Research J. (In Press)

Two processes for producing wash-wear cotton fabrics by treatment with formaldehyde are described. One of these processes consists of reacting the fabric in a wet swollen condition in an aqueous solution of formaldehyde and hydrochloric acid. This fabric has good wet wrinkle recovery only and is therefore referred to as the Form-W process. It has good strength retention and is suitable for line-drying after washing. The other process consists of reacting the fabric in a wet, partially swollen condition in a solution of acetic acid, water, hydrochloric acid, and formaldehyde. It is referred to as the Form-D process because the fabrics have good dry (as well as wet) wrinkle recovery. These fabrics are suitable for either line-drying or tumble-drying. It is very important in both processes that the fabrics be well mercerized if adequate strength is to be retained. Softeners also improve the tearing strength. Laboratory and pilot-plant applications are described, and data on the physical properties of the fabrics presented. Reaction rates of the Form-D process at various temperatures are given. Also, preliminary work on other solvents and catalysts is discussed briefly. Crosslinking with formaldehyde at various degrees of fiber swelling is discussed briefly from a theoretical standpoint.

EFFECTS OF TENSION DURING RESIN TREATMENT ON PHYSICAL PROPERTIES OF COTTON FIBERS AND YARNS

Orr, Rollin S.; Burgis, Albert W.; and Grant, James N.
Textile Research J. (In Press)

The effects of tension during resin treatment on the physical properties of yarns and fibers from the yarns were investigated on several cotton samples. The resins applied were of the melamine-formaldehyde and dimethylolethyleneurea types. The strength loss from slack treatments was the result of crosslinking of the internal fiber elements in unfavorable positions for stress equilization. Acid degradation was a factor in certain treatments. Low tensions during treatment were sufficent to prevent much of the strength loss. As elongation was reduced by tension, the energy to rupture was not greatly changed. The selection of high strength, high elongation cottons, and premercerization increased the toughness. Tension before and during drying, followed by low tension during the cure, minimized the losses of toughness in yarns.

WRINKLE- AND MUSS-RESISTANT FINISH FOR COTTON USING FORMIC ACID COLLOID OF METHYLOLMELAMINE

Berard, W. Norbert; Leonard, Ethel K.; and Reeves, Wilson A.
Am. Dyestuff Reptr. (In Press)

Cotton fabric has been made wrinkle resistant by treating with a freshly prepared formic acid colloid of methylolmelamine. The method of application of the freshly prepared acid colloid is essentially the same as used for resin finishing; that is, pad, dry, and cure. The main difference is the use of formic acid in the treating solution to produce a colloid and to serve as the catalyst. Curing is accomplished at 140°C. Print cloth finished with the acid colloid of methylolmelamine at about 7% resin add-on has good wrinkle and muss resistance. Finished fabrics compare favorably with fabrics finished by a conventional process as to breaking strength, tear strength, wrinkle recovery, chlorine damage, and nitrogen retention, after 5, 10, and 20 wash and tumble drying cycles.

Although acid colloid finished print cloth yellows less when treated with strong sodium hypochlorite solutions than conventional trimethylolmelamine finished cottons, the finish is not recommended for white goods. Microscopical studies made on treated fibers show the resin deposited primarily in the outer portion of the cell wall of the cotton fiber, and the innermost section of the fiber responded to cupriethylenediamine hydroxide in a manner similar to that of cotton having small amounts of crosslinked cellulose.

FORMIC ACID COLLOID FOR METHYLOLMELAMINE RESIN FINISH OF COTTON FOR WRINKLE- AND MUSS-RESISTANCE: PRELIMINARY COST STUDY

Decossas, E. M.; McMillan, O. J., Jr.; Berard, W. N.;
Reeves, W. A.; Pollard, E. F.; Patton, E. L.
Am. Dyestuff Reptr. (In Press)

Investment and operating costs for a hypothetical plant are reported for the application of the formic acid colloid of methylolmelamine resin finish to cotton to impart wrinkle- and muss-resistance. The process is conventional, consisting of padding, drying, and curing, and is also versatile in that variation of the composition of the treatment solution and of resin add-on produces cotton products exhibiting differing desirable quality characteristics for various end uses. Cotton fabrics finished to a 7 percent resin add-on by this technique have good wrinkle- and muss-resistance; and have good resistance to chlorine scorch damage even after repeated laundering.

The cost study is based on finishing 80 x 80 cotton print cloth, 50 inches wide, 3.12 linear yards per pound, to a 7 percent resin add-on, by continuous operations in a hypothetical plant, at a processing rate of 120 yards per minute. It is estimated that processing cost ranges from 3.7 cents per linear yard at an annual production of 60.5 million yards to 5.1 cents per linear yard at an annual production of 14.4 million yards. Alternatively expressed, the costs range from 2.7 to 3.7 cents per square yard, or from 10.8 to 14.8 cents per pound of product, exclusive of the cost of cotton used and without profit.

STUDIES ON THE CROSSLINKING OF COTTON CELLULOSE: PART II. MICROSCOPICAL OBSERVATIONS

Tripp, Verne W.; Moore, Anna T.; and Rollins, Mary L.
Textile Research J., Accepted
(For Part I see No. 2051, Page 11)

The response of crosslinked cotton fibers to beating in water, enzymatic hydrolysis, and exposure to cellulose solvents has been examined by electron microscopy. Crosslinking by primary bonds between cellulose chains confers stability of the cellulose structure toward mechanical and chemical attacks of these types. Crosslinks introduced when the fiber is in the unswollen state are more effective in stabilizing the cellulose than those established when the cotton is swollen. The significance of the observations is discussed.

PUBLISHED

2072. CHANGES IN THE PROPERTIES OF PARTIALLY PHOSPHONOMETHYLATED COTTON CAUSED BY CROSSLINKING PRIOR TO PHOSPHONOMETHYLATION

Hobart, Stanley R., Drake, George L., Jr.; and Guthrie, John D.
Am. Dyestuff Reptr. 50, 30-34. 1961

Crosslinking the cellulose of cotton fabrics with dichloropropanol prior to phosphonomethylation with disodium chloromethyl phosphonate made possible a higher degree of substitution with loss of fabric structure. Crosslinked cotton print cloth and sheeting were phosphonomethylated (both once and twice). The degree of substitution increases over non-crosslinked phosphonomethylated fabrics were approximately 25 and 50%, respectively. Flame resistance, cation exchange capacity, resistance to microbiological degradation, and moisture content increased approximately in proportion to the increase in degree of substitution. Furthermore, prior crosslinking produced fabrics with significantly less stiffness, and nearly normal air-permeability. Pilot plant equipment was used for all of the treatments.

2051. SOME STUDIES ON THE CROSSLINKING OF COTTON CELLULOSE.
PART I: CHEMICAL AND PHYSICAL ASPECTS
Perkerson, Fred S.; Reeves, Wilson A.; and Tripp, Verne W.
Textile Research J. 30: 944-954. 1960

The currently-used types of crosslinking agents and some of their effects on cotton are discussed. The primary weaknesses or shortcomings of the finishes achieved with these agents are pointed out. It can be concluded either that the ideal crosslinking agent for cotton has not been found or that the best technique of application has not yet been discovered. However, considerable success is had in producing minimum-care fabrics by the use of many crosslinking agents, especially the N-methylol type. Although crosslinks through primary covalent bonds are largely responsible for the observed physical changes in crosslinked cotton fiber, secondary valence crosslinks also may produce significant changes, some of which appear to be rather durable. Evidence is presented which indicates that polymer formation produced concurrently with crosslinking improved the over-all minimum-care properties of cotton fabric, although polymer formation alone does not improve the wrinkle recovery. Crosslinking agents which are capable of forming thermosetting resins generally are required in somewhat larger add-ons to produce wrinkle recovery angles in the practical range than are those agents that do not form thermosetting resins. The extent of permanent set is related to the extent of crosslinking. Moisture regain and density of the crosslinked cottons are more dependent upon the state of fiber swelling at the time of crosslinking than upon the extent of crosslinking.

2049. IMPARTING WRINKLE RESISTANCE TO COTTON WITH 1,1'-CARBONYLBIS-AZIRIDINE
Chance, Leon H.; Perkins, Rita M.; and Reeves, Wilson A.
Textile Research J. 30, 918-926. 1960

Cotton print cloth and broadcloth were made wrinkle resistant by treating with carbonylbisaziridine (CBA) by the conventional process of padding, drying, and curing. Data are presented for fabric treated with and without the use of an acid catalyst, zinc fluoborate. An after-treatment with Primenit VS emulsion increased the crease recovery angle. Monsanto crease angles of over 300° were obtained with resin add-ons as low as 4.5%. In general the Elmendorf tearing strength was reduced 40-50%. This was improved somewhat by the use of a softener. The finish was quite stable to home laundering. When no catalyst was used in the solution, a reduction in breaking strength was observed after the chlorine bleach and scorch. When freshly prepared CBA solutions containing zinc fluoborate were used, there was no reduction in the strength of fabric after the chlorine bleach and scorch. However,

all fabrics containing the finish were discolored in the scorched areas.

2030. REACTION OF COTTON CELLULOSE WITH EPOXIDES IN THE PRESENCE OF ACID CATALYST

McKelvey, John B.; Webre, Beverly G.; and Benerito, Ruth R.
Am. Dyestuff Reptr. 49, 804-809. 1960

Ease of reaction of cellulose and various available epoxides in the presence of hydrogen ion catalysts and Lewis acids is considerably different than results obtained previously with base catalysis. Only a few monoepoxides add to cotton in the presence of cold dilute mineral acid; in the case of ethylene oxide, the addition was about 10% of that obtained in the presence of hydroxyl ion. Diepoxides tested were also rather inert in the presence of hydrogen ion; the best (butadiene diepoxide) adding to cotton only about half as much as the best monoepoxide (butadiene monoepoxide). Under conditions of cationic catalysis (Lewis acid), especially with zinc fluoborate, it has been found that only two monoepoxides react with cotton. In the case of diepoxides, some of which are known to be very active, it was shown that crease resistance may be imparted to cotton at very low add-on by butadiene diepoxide, and that several diepoxides tested while producing good enough add-on failed to impart dry crease resistance. When epoxides which are known to add to cotton by zinc fluoborate catalysis are reacted under optimum conditions of base catalysis, it was found that the base-catalyzed samples exhibited only good wet crease resistance.

2012. FABRIC STRUCTURE AND WASH-AND-WEAR
/Influence of Fabric Structure on Tear Strength of Resin-Treated Cotton Fabrics/

Stavrakas, E. James; and Platt, Milton M.
Contractor: Fabric
Research Laboratories, Inc., Dedham, Mass.
Textile Inds. 124, No. 10, 141-160. 1960

Analytical determination of the causes of the loss in tear strength exhibited by the principal types of commercial cotton fabrics following resin treatment showed the following: That large losses in tearing strength result from the preparatory finishing processes, i.e., desizing, scouring, bleaching, and dyeing; that scouring was largely responsible for this loss in tear strength; that commercially resinated fabrics may exhibit either losses or improvements in tear strength; that the fabric structural types exhibiting significant reductions in tear strength also had large losses in yarn strength and slight changes in the number of threads rupturing per peak; but that the structural types exhibiting higher tear strength displayed significant increase in the number of threads rupturing per peak and considerably less reduction in yarn tensil strength.

Theoretical and empirical analysis indicate that the tear strength of cotton fabrics may be improved through structural changes which increase the mobility of the yarns in the plane of the fabric, by increasing yarn strength, or by a combination of these factors. Improvements in tear strength may be obtained through one or more of the following: use of weaves with longer floats, use of more open textured fabrics, use of opposite twists in warp and filling yarns, and use of coarser, stronger yarns.

2006. COTTON FABRIC WITH WET WRINKLE RECOVERY PRODUCED WITH FORMALDEHYDE WITHOUT EXCESSIVE STRENGTH LOSS

Reeves, Wilson A.; Perkins, Rita M.; and Chance, Leon H.
Am. Dyestuff Reptr. 49, 639-644. 1960

The crosslinking of cotton fabric with formaldehyde while in a water swollen state was studied with the object of producing by a practical method a fabric having good wet wrinkle recovery without excessive degradation of fabric strength. This was accomplished in a pilot-plant by padding the fabric through an aqueous solution of formaldehyde and hydrochloric acid and allowing to react in the wet state at room temperature by (1) remaining on the roll, (2) plaiting into a box (referred to as a J box process). After the required length of time (five to 20 minutes) the fabrics were given an alkaline process wash, and dried. Bleached mercerized fabric was found to be more suitable than bleached fabric in the process because a given degree of wet wrinkle recovery was acquired at a faster rate and a lower level of combined formaldehyde. Also the final tear strength, and breaking strength, which were much greater with the mercerized fabric, were well within a practical range. Fabrics treated by this process had good wet wrinkle recovery only, and would be of practical use only when line dried. Preliminary experiments indicated that cotton fabrics which were reacted to a low degree with formaldehyde and then topped with a small amount of crease-proofing resin (in the range of 2.5%) such as a triazone, had as much initial wet and dry wrinkle recovery as was obtained with about 5% of the same resin on the original cotton fabrics.

1986. STUDIES OF WRINKLE RESISTANCE FINISHES FOR COTTON TEXTILES. II--EFFECT OF STORAGE ON PROPERTIES OF "WASH-AND-WEAR" COTTONS

Reid, J. David; Reinhardt, Robert M.; Kullman, Russell M. H.; and Arceneaux, Richard L.
Am. Dyestuff Reptr. 49, 527-531. 1960

The properties of some "wash-and-wear" cotton textiles are adversely affected by prolonged storage prior to reaching the consumer. In the present study, the effects of storage of fabrics treated with five types of N-methylol finishing agents were investigated. Chlorine resistance, breaking and tearing strength, and wrinkle resistance were determined. The process was after curing was found to be a critical factor affecting the

durability of desirable properties of the finished fabric to storage. Particularly vulnerable to degradation by storage was the chlorine resistance of fabric treated with dimethylol ethyleneurea and the wrinkle resistance of fabric treated with a pyrimidinone derivative when the fabric was not afterwashed. Cotton treated with a triazone or melamine derivatives also showed increased chlorine damage when not washed before storage.

1983. EFFECTS OF CROSSLINKAGE IN WRINKLE-RESISTANT COTTON FABRICS
Frick, J. G., Jr.; Andrews, B. A. Kottes; and Reid, J. David
Textile Research J. 30, 495-504. 1960

The crosslinking of cellulose in cotton fabric with three wrinkle-resistance finishing agents has been investigated. The three agents used were dimethylol urea, dimethylol ethyleneurea, and formaldehyde. With all three compounds, maximum crease recovery angles are obtained with a substitution of 0.04-0.05 crosslinks per anhydroglucose unit. As crease recovery angles increase, the elongation and permanent set caused by an 8-kg. tensile load on a 1-in. fabric strip decreases. Permanent set approaches zero near 135° warp crease recovery. Changes in the load-elongation curves indicate elastic modulus increases as crease recovery angles increase. Treatment of fabric to the same crease recovery angles with the three compounds reduces tearing and breaking strengths to different extents. The differences are due to different amounts of cellulose degradation caused by the acidic catalyst systems and not to the structure of the crosslinks.

1981. STUDIES OF WRINKLE RESISTANCE FINISHES FOR COTTON TEXTILES. I--RELEASE OF FORMALDEHYDE VAPORS ON STORAGE OF WRINKLE-RESISTANT COTTON FABRICS
Reid, J. David; Arceneaux, Richard L.; Reinhardt, Robert M.; and Harris, James A.
Am. Dyestuff Reptr. 49, 490-495. 1960

The evolution of formaldehyde from fabrics treated for wrinkle resistance with several N-methylol finishing agents has been studied and the amounts of formaldehyde released on storage have been quantitatively determined. The effects of processing variables, such as time and temperature of cure, amount and type of finishing agent applied, type of catalyst used and the process wash following curing, have been investigated as related to the amount of formaldehyde produced. Also studied were the principal variables in storage, time and temperature. The process wash after curing was found to have the greatest effect in reducing the amount of formaldehyde evolved from treated fabrics. It was shown experimentally that this was due to removal of residual catalyst by the wash. An accelerated storage test was used in the determination of formaldehyde evolution and the results were found to have excellent correlation with those of prolonged storage under normal conditions.

1977. EFFECT OF FABRIC STRUCTURE ON PROPERTIES OF WRINKLE-RESISTANT COTTONS

Arceneaux, Richard L.; Reid, J. David; and Schultz, E. Fred, Jr.
Textile Inds. 124, No. 9. 138-146. 1960
Also, condensed version entitled "Wrinkles and Weaves"
Textile World 110, No. 7. 121-122. 1960

Twenty-three cotton fabrics varying in one or more structural parameters were treated with a wrinkle-resistance agent, dimethylol ethyleneurea (DMEU), and also with the agent in a formulated treatment. The effects of the structural variables on crease recovery angle, tearing strength, breaking strength, and resin distribution were studied. Interactions of fabric properties with fabric direction necessitated a study of fabric properties on each direction rather than on the average or sum of both directions. The treatments tended to narrow the spread of crease recovery angle found in the untreated fabrics, bringing the crease recovery angle of each fabric closer to the typical of the treatment. The use of fancy rather than plain weaves caused an improvement in tearing strength properties. The use of coarser yarns also improved tearing strength properties. Distribution of resin content between warp and filling yarns was affected by variations in yarn twist multiplier. Breaking strengths of the various fabrics were not greatly affected by the structural variations in the fabrics.

1942. CROSS-SECTIONAL STUDY OF CHEMICALLY TREATED COTTON FIBERS

Rollins, Mary L.; Moore, Anna T.; and Tripp, Verne W.
Intern. Conf. Electron Microscopy, Berlin, Germany, 1958
Proc. 4, 712-715. 1960

Ultrathin cross sections of cotton fibers mounted on carbon-coated specimen grids have been subjected to the action of reagents which dissolve cellulose. This technique has permitted following with the electron microscope changes in the behavior of cotton cellulose as a result of crosslinking, substitution and polymer impregnation. Using cupriethylenediamine hydroxide as a swelling agent, cross sections of untreated cotton fibers have been observed to dissolve whereas sections of fibers which have been treated with appropriate chemical crosslinking agents swell and only partially dissolve. Furthermore, in cases where there has been extensive lateral crosslinking of the cellulose within the fiber there is phenomenally great swelling of the cross section without any separation or rupture of morphological elements. In fibers in which previously introduced monomers have been polymerized within the fiber the swelling of the cross section is considerably restricted. Similar observations on fiber fragments complement these findings.

1935. ALDEHYDE-ETHYLENIMINE REACTION PRODUCTS AS FINISHES FOR COTTON FABRICS

Chance, Leon H.; Perkins, Rita M.; and Reeves, Wilson A.
Textile Research J. 30, 305-311. 1960

In an exploratory study the reaction products of ethylenimine with four aldehydes were evaluated primarily as wash-wear finishes for cotton fabric. Two dialdehydes, glyoxal and glutaraldehyde, and two alpha, beta-unsaturated monoaldehydes, acrolein and croton-aldehyde, were used in the study. One of the new finishing agents, the reaction products of acrolein and ethylenimine, imparted good wrinkle recovery to cotton fabric.

1922. COTTON CROSSLINKED AT VARIOUS DEGREES OF FIBER SWELLING

Reeves, Wilson A.; Perkins, Rita M.; and Chance, Leon H.
Textile Research J. 30, 179-192. 1960

Wet and dry density measurements were made on a wide variety of wash-and-wear cotton fabrics and some interpretations of these values are given. Methylenated cotton was selected for more extensive fundamental study because of the simplicity of the formaldehyde crosslink and the fact that formaldehyde can be reacted with cotton under greatly different conditions. Formaldehyde was used to crosslink cotton print cloth at various degrees of fiber swelling by reacting in systems ranging from completely aqueous to anhydrous using hydrochloric acid as catalyst. The amount of water in the system at the time of reaction is related to the extent of fiber swelling. The extent of swelling at the time of crosslinking and the extent of crosslinking influence wet density, which may be considered to be a measure of the water swellability of a fiber. Crosslinking tends to fix or stabilize a fiber in a given state of swelling; the stabilization is more effective when the crosslinking is done in nearly anhydrous systems. The state of swelling at the time of crosslinking modifies moisture regain, water of imbibition, dyeability, and wet and dry wrinkle recovery. There seems to be an optimum water content in the reaction system at the time of crosslinking for maximum dry and wet wrinkle recovery. As the water content in the reaction system is increased beyond the optimum, the amount of dry wrinkle recovery becomes much less than wet wrinkle recovery. This phenomenon leads to vastly different wash-and-wear ratings of fabric. A mechanism is presented diagrammatically to explain wet and dry wrinkle recovery. The mechanism is based upon hydrogen-bond crosslinks, covalent-bond crosslinks, and the position of the two types of crosslinks.

1723. FORMIC ACID COLLOID OF METHYLOLMELAMINE AS A WEATHER AND ROT RESISTANT FINISH FOR COTTON

Berard, W. Norbert; Gautreaux, Gloria A.; and Reeves, Wilson A.
Textile Research J. 29: 126-33.

A study of application techniques of methylolmelamine resins to cotton has revealed a practical method for producing cotton fabric with outstanding rot resistance and improved weather resistance. This method is based upon the use of an acid colloid of methylolmelamine. The method of application of the colloid is the same as that conventionally used for resin finishing. The main difference is the use of formic acid to produce a colloid. Photomicrographs show that methylolmelamine colloids (1-4 hr. old) are deposited in the outer portion of the cell wall of the fiber, whereas the conventional process deposits resin about halfway through the cell wall. Only these fresh colloids give outstanding rot resistance. Cotton fabric finished with the acid colloids compares favorably with partially acetylated, fully acetylated, cyanoethylated, and other treated fabrics which are known to have excellent resistance to rot. The crease recovery angles for the colloid finished fabrics were quite encouraging. Several acids were evaluated for colloid production; formic was the only acid which produced suitable colloids. Small amounts of the metals become tenaciously bound in the fabric, and some of the metals alter outdoor weathering and other properties of the fabric.

1947. AN APPROACH TO AUTOMATION OF A WRINKLE RECOVERY TESTER

Sloan, Julia M.; Haydel, Chester H.; Schultz, E. Fred, Jr.; and Weller, Heber W., Jr.
Am. Dyestuff Reptr. 49, 302-304 (1960)

This paper describes the electronically operated mechanisms which have been employed in an initial approach toward automation of the wrinkle recovery tester used in the vertical strip method (Monsanto) for measuring wrinkle recovery angles. Essentially it provides means for automatically adjusting the position of the specimen during the recovery period by means of a photoelectric cell. It also times and stops the test at the end of the test interval.

ECONOMIC EFFECTS OF WASH AND WEAR COTTON (Unnumbered)

Barlow, Frank D., Jr.
U. S. Dept. Agr., Agr. Marketing, 4 (9): 3. 1959

The importance of wash-wear apparel and household goods, the main uses for resin-treated cotton, can be measured in both dollars and cents and in greater freedom from housework. In addition to laundering ease, resin-treated cotton resists crushing and wrinkling better than the untreated. It doesn't shrink as much, and holds its shape better. These advantages have brought about an increasing demand for wash-wear cotton fabrics. In 1958 about two billion yards of cotton textiles were treated with easy-care finishes, with 3-1/2 to 4 billion yards predicted for 1960, or about two-thirds of the broad woven goods now being finished for apparel and household uses.

1915. LOCATION OF AREAS OF REACTION IN CHEMICALLY TREATED COTTON CELLULOSE

Tripp, V. W.; Moore, A. T.; deGruy, I. V.; and Rollins, M. L.
Textile Research J. 30, 140-147. 1960

The modification of cotton cellulose by environmental conditions or by chemical treatments for specific end uses ordinarily occurs at submicroscopic levels. Observations with the electron microscope have proven to be useful in delineating regions of the fiber cellulose affected by a variety of treatments, including dyeing, mercerization, acid and enzymatic hydrolysis, derivative formation, and resin impregnation. The appearance of fiber fragments treated in these ways or derived from treated fibers is illustrated and discussed.

1890. DEVELOPMENT OF METHODS OF RESIN APPLICATION TO COTTON GARMENTS FOR DURABLE CREASING AND WRINKLE RESISTANCE BY USE OF STANDARD DRYCLEANING EQUIPMENT

Wiebush, Joseph R.; Graham, Robert T.; and Loibl, Fred.
Contractor: National Institute of Drycleaning, Silver Spring, Md.
Natl. Inst. Drycleaning Publ. F-19, 4 pp. December 1959

A research program has been carried out by the National Institute of Drycleaning to determine the feasibility of the application of finishes to impart wrinkle resistance and durable creases to cotton garments using ordinary drycleaning plant equipment. It has been shown that such finishes can be successfully applied to many types of garments, particularly shirts, blouses, skirts, trousers, and jackets. In general, medium or light-weight plain-weave fabrics, such as print cloth, ginghams, and broadcloths respond better than heavier weight fabrics. The finishing procedure consists of applying a resin formulation, extracting in a centrifuge to control the pickup, shaping the garment with hot-head presses and hand equipment, and completing the cure in a hot air drying cabinet. The garment is then washed and tumble dry. The effect of variations in this procedure are reported. Preliminary cost data on the process have been calculated. This type of finishing is best suited for use in conjunction with garment manufacturing but could be useful as a supplementary service by many drycleaners, especially during the slack season.

1838. REACTION OF EPOXIDES WITH COTTON CELLULOSE IN THE PRESENCE OF SODIUM HYDROXIDE

McKelvey, John B.; Webre, Beverly G.; and Klein, Elias
Textile Research J. 29, 918-25. 1959

The reactions of various commercially available epoxides with cotton cellulose in the presence of sodium hydroxide have been investigated at 25°, 50°, and 95°C. The effects of solvents and alkali content of the yarn steep on the epoxy add-on have been studied.

1835. THE PREPARATION AND PROPERTIES OF PARTIALLY PHOSPHONOMETHYLATED COTTON

Hobart, Stanley R.; Drake, George L., Jr.; and Guthrie, John D.
Textile Research J. 29, 884-80. 1959

Cotton fabrics have been phosphonomethylated on commercial scale textile finishing equipment and the properties of the treated fabrics evaluated. At sufficiently high phosphorus contents the fabrics have a permanently starched feel and improved wet crease resistance. Good flame resistance is observed for the ammonium salt form of the fabrics. The treated fabrics are more hydrophilic than the controls. Stability of the phosphorus content to alkaline treatments is very high. Considerable improvement over untreated cotton is shown by the copper salt form in regard to microbiological deterioration.

1831. PARTIALLY CARBOXYMETHYLATED COTTON AS AN INTERMEDIATE FOR FURTHER CHEMICAL MODIFICATION

Reinhardt, Robert M.; Reid, J. David; Fenner, Terrence W.; and Mayne, Ruth Y.
Textile Research J. 29, 802-10. 1959

Cotton can be etherified by treatment with chloroacetic acid and sodium hydroxide solution. The product, a partially carboxymethylated cotton with the fibrous nature of the original, has a number of properties valuable for textile and other uses. In addition, the etherified cotton bears two reactive functional groups--hydroxyl and carboxyl--capable of further chemical modification. The rate and extent of many reactions with the etherified cotton are greater than those of ordinary cotton similarly treated. Further modifications of this cotton derivative are discussed. Among these are salt formation for the preparation of bactericidal and fungistatic finishes, reactions for the introduction of cyanoethyl, carboxyethyl, and graft polyester groups, reaction with epoxy and aziridinyl compounds, and oxidation for the preparation of alkali-soluble textile products.

1828. MICROSCOPICAL EVALUATION OF COTTON FINISHING TREATMENTS

Rollins, Mary L.; deGruy, Ines V.; Tripp, Verne W.; and Moore, Anna T.
Am. Soc. Testing Materials "Symposium on Microscopy"
Spec. Tech. Publ. No. 257, 153-65. 1959

Laboratories of the textile industry expend considerable time and money attempting to improve wrinkle resistance of cotton fabrics. This paper defines the function of the microscopist in these research efforts which are directed both toward empirical formulation for commercial application and fundamental investigations of reaction mechanisms. After explaining the architecture of the cotton fibers, the authors turn to the different types of cotton finishing-purification of the fiber for removal of noncellulosic substances, and physical modification of the fiber through treatment with chemicals which, by altering the crystalline structure

of the fiber, permanently change its properties. The paper considers also additive finishing through coatings or impregnants, and finishing through chemical modification of the cellulose which produces new substances without changing the form of the fiber. The paper discusses techniques of staining, cross-sectioning, swelling and dissolution; refractive index determination in optical microscopy; and techniques of replication, fragmentation, ultrathin sectioning, swelling and dissolution in specimen preparation for electron microscopy. It is supplemented by micrographs illustrative of optical and electron microscope observations.

1812. DURABLE CREASES PRODUCED IN COTTON AND RESIN FINISHED COTTON FABRICS BY AN ALKALI PROCESS

Reeves, Wilson A.; and Mack, Charles H.
Am. Dyestuff Reptr. 48 (21) 43-46, 50. 1959

A process is described for imparting durable creases to cotton fabrics. The process utilizes mercerization strength alkali solutions and is applicable to most types of resin-treated and cross-linked fabrics, as well as to unfinished cotton fabrics. Creases are produced by moistening the area to be creased with alkali solution, then ironing-in the creases with a hot iron. Creases produced by the process remain sharp after many launderings.

1807. CHLORINE-RESISTANT BLENDS OF TRIAZONE AND UREA DERIVATIVES FOR WRINKLE-RESISTANT COTTON FABRICS

Frick, J. G., Jr.; Arceneaux, Richard L.; Reinhardt, Robert M.; and Reid, J. David
Am. Dyestuff Reptr. 48, (15): 29-31, 36. 1959

Wrinkle-resistant finishes prepared from blended methylol derivatives of ethyl triazone and urea are described. Some of these finishes are not subject to damage from retained chlorine and possess economical and other advantages of over the triazone finishing agents used alone.

1845. WRINKLE-RESISTANT COTTON WITH DURABLE CHLORINE RESISTANCE

Frick, J. G., Jr.; Murphy, Paul J., Jr.; Reinhardt, Robert M.; Arceneaux, Richard L.; and Reid, J. David
Am. Dyestuff Reptr. 48, (18) 37-40. 1959

Finishing agents for producing wrinkle-resistant and "wash-and-wear" cotton fabrics have been developed, using mixtures of dimethylol ethyleneurea and a formal of a polyhydroxy compound. These agents give good wrinkle resistance and chlorine resistance, both of which are durable to repeated launderings.

1819. IMPARTING WET CREASE RESISTANCE TO COTTON FABRICS BY CROSSLINKING WITH AQUEOUS FORMALDEHYDE
Guthrie, John D.
Textile Research J. 29, 834-36. 1959

The wet crease recovery of cotton fabrics was improved by crosslinking with aqueous formaldehyde using acidic catalysts. With formic acid or ammonium chloride a solution temperature of about 80°C. was required for 6 hours to obtain good wet crease recovery, but with sulfuric acid, 3 hours at room temperature (28°C) gave satisfactory results. Dry crease recovery was not improved significantly. Strength losses appeared to be in the range tolerated for wash-and-wear fabrics.

1833. PHENOLIC-FORMALDEHYDE RESINS AS FINISHING AGENTS FOR COTTON FABRICS
Chance, Leon H.; Perkerson, Fred S.; and McMillan, Oscar J., Jr.
Textile Research J. 29, 558-64. 1959

Cotton fabric was treated with phenolic-formaldehyde resins and seven halogenated phenolic-formaldehyde resins and the properties of the various samples evaluated. The fabric was treated by padding it through a solution of phenol in alkaline aqueous formaldehyde, drying, and curing in an oven. All of the fabrics had improved rot resistance and crease recovery. The halogenated phenols did not seem to have any advantage over plain phenol in imparting rot resistance to cotton fabric. Fabrics treated with m-chlorophenol and m-bromophenol had the highest crease recovery.

1716. CHEMICAL AND PHYSICAL EFFECTS OF FINISHING COTTON WITH METHYLOL DERIVATIVES OF ETHYLENEUREA
Frick, J. G., Jr.; Kottes, Bethlehem A.; and Reid, J. David
Textile Research J. 29: 314-22. 1959

A study has been made of the mechanism by which cotton is made wrinkle resistant by dimethylol ethyleneurea. Estimates are made of the length of crosslinks and the relative effect of several factors, including processing conditions, crosslinkage, and acid damage, on the loss in strength of the fabric on treatment. Various methylol derivatives of ethyleneurea were used in the study of the effect on chemical and physical properties. It has been demonstrated that the methylol derivatives of ethyleneurea react chemically with cellulose under the influence of acidic catalysts. Metallic salts are most efficient in promoting this reaction. Both monofunctional and difunctional compounds react with the cellulose. The latter, however, cause crosslinkage, which effects marked changes in the physical properties of the treated cotton fabric, particularly wrinkle resistance. Dimethylol ethyleneurea forms crosslinks averaging

1.4-1.5 ethyleneurea residues in length. These residues are connected by methylene groups. The effects of variations in catalyst, composition of the finishing agent, and curing conditions are shown. Relation of the findings to practical finishing is discussed.

1724. IMPARTING CREASE RESISTANCE AND CREASE RETENTION TO COTTON WITH APO

Drake, George L., Jr.; and Guthrie, John D.
Textile Research J. 29: 155-64. 1959

Cotton fabric (80 x 80) was made crease resistant by processing with APO, tris(1-aziridinyl) phosphine oxide. Application was made by wetting the fabric with an aqueous solution of APO containing catalytic quantities of zinc fluoborate, squeezing out the excess with squeeze rolls, drying for 4 min. at 80-80°C., and curing for 4 min. at 140°C., followed by a good wash. An after-treatment with a 1% Primenit VS3 solution increased the crease recovery angle. Monsanto crease angles of from 250° to more than 300° (warp + fill) have been obtained, depending on the resin add-on. The crease recovery is not changed much by home laundering. A loss of 30% in the Elmendorf tearing strength was observed with samples with over 300° crease recovery angles. Creases durable to washing were obtained by drying the fabric with a hand iron followed by an oven cure. The resin could not be stripped from the fabric by methods that are effective with other resins. In addition to imparting crease resistance, this resin also imparts some degree of flame resistance.

1733. PHOSPHONOMETHYLATION OF COTTON

Drake, George L., Jr.; Reeves, Wilson A.; and Guthrie, John D.
Textile Research J. 29: 270-75. 1959

A new phosphorus-containing ether of cotton cellulose has been made by reacting cotton with disodium chloromethylphosphonate in the presence of sodium hydroxide. The degree of substitution can be varied widely by varying the concentration of the phosphonomethylating agent and of the sodium hydroxide and by varying the time and temperature of the cure. Fabric containing up to 3.96% phosphorus was produced by giving the fabric a double treatment. At this degree of substitution the textile was quite soluble in water. Up to 2.25% phosphorus was introduced by a single treatment. Generally, the modified cotton becomes water-soluble when about 2.0% phosphorus is introduced. A permanently starched effect is produced with phosphorus contents of approximately 1%. At this degree of substitution there is little or no change in the breaking strength of the fabric. Modified cotton containing between 1% and 2% phosphorus has greatly increased swelling properties in water. The phosphonomethyl cotton is produced as the sodium salt. It can be converted to the free acid form by soaking

or rinsing in dilute mineral acid. The pK_a values for the first and second ionizations are 1.9 and 7.0, respectively. When in the ammonium salt form, the phosphonomethylated cotton has good flame resistance.

1732. PERMANENT SET IN COTTON AND THE PHYSICAL MECHANISM OF WRINKLE RESISTANT TREATMENTS

DuPre', A. Mason, Jr.
Textile Research J. 29: 151-55. 1959

The mechanism of stretching of the cotton fiber is essentially different from that of any other textile fiber, either natural or synthetic. Due to the fact that there are frequent reversals in the direction of spiralling of the fibrils, and the fact that the reversals in all of the growth layers, beginning with about the second, occur at approximately the same cross-sectional points along with length of the fiber, the cotton fiber stretches by actually untwisting. More specifically, each segment between two points of reversal untwists, with the segment on one side of a reversal untwisting in a direction opposite to that of the segment on the other side. Hence, it is deduced that a set of torques is set up along the length of the fiber when it is stretched. Further, the average angle of the fibrils to the fiber axis is of a magnitude such that the amount of elongation observed in cotton fibers can be accounted for by this untwisting, without assuming any elongation in the fibrils themselves. Studies made with the electron microscope show that on extreme swelling the growth layers are separated, with little evidence of swelling of the fibrils themselves. Also, when cotton fibers are mechanically broken up in water in a Waring blender, there is evidence of strong lateral cohension between fibrils. These facts have important implications regarding (1) the nature of permanent set in cotton fibers with respect to cotton's unique structure, and (2) the mechanism by which crosslinking agents reduce the elongation and increase the elastic recovery of cotton fibers. A brief review of some phases of current theories in this field is given, together with some observations pertinent to their validity. It is proposed that there is at least some relative movement between growth layers, that this relative movement may contribute significantly to permanent set, and that crosslinking between growth layers may contribute appreciably to wrinkle resistance.

1738. TETRAHYDROPYRIMIDINONE DERIVATIVES FOR NONCHLORINE RETENTIVE, WRINKLE-RESISTANT COTTON FABRICS

Frick, J. G., Jr.; Kottes, Bethlehem A.; and Reid, J. David
Am. Dyestuff Reptr. 48: 23-25. 1959

A wrinkle-resistant and "wash-and-wear" finish for cotton fabrics based on the dimethylol derivative of tetrahydro-5-hydroxy-2(1H)-pyrimidinone has been developed which shows several advantages over currently used finishes. The finishing agent is structurally

similar to dimethylol ethyleneurea and produces a finish with similar properties. It causes no chlorine retention, however, and this property is retained after repeated hot, alkaline launderings. Also, it does not yellow on heating as the triazone finishes often do and leaves no residual odor. It is, however, susceptible to hydrolytic removal by acidic conditions below pH4, which renders it vulnerable to overly acidic or hot souring conditions. Although in this vulnerability it is similar to finishes produced from dimethylol ethyleneurea, the pyrimidinone finish loses less chlorine resistance than the ethyleneurea finish under acidic conditions, prior to complete removal.

1815. IMPARTING WRINKLE RESISTANCE TO COTTON FABRICS WITH TRIAZONE DERIVATIVES

Reid, J. David; Frick, J. G., Jr.; Reinhardt, Robert M.; and Arceneaux, Richard L.
Am. Dyestuff Reptr. 48: P81-P90. 1959

The need for a finishing agent which will give good wrinkle resistance and other improved textile properties to cotton, and which will be partially unaffected by laundering, particularly by chlorine bleaching and the highly alkaline and acid conditions encountered in commercial laundering, led to an investigation of some "triazone" derivatives. Since compounds of this type are coming into commercial use in this country and in Europe, an evaluation of the properties is in order. The methylol derivatives of 5-substituted tetrahydrotrizinone have been investigated as finishing agents for imparting wrinkle resistance and "minimum-care" properties to cotton fabrics. The preparation and properties of some of these agents and the properties of the finished fabrics are described. These triazones are potentially inexpensive and may be applied to fabric by conventional procedures. The properties of the finished fabrics are compared with those of fabrics finished with some other commercial wrinkle-resistance agents and the advantages and disadvantages of each are discussed.

1643. AFTERMERCERIZATION OF WRINKLE-RESISTANT COTTONS FOR IMPROVED STRENGTH AND ABRASION RESISTANCE

Reinhardt, Robert M.; Kullman, Russell M. H.; Moore, Harry B.; and Reid, J. David
Am. Dyestuff Reptr. 47: 758-64. 1958

A previous study showed that improvement in tearing strength and abrasion resistance was produced by aftermercerization of cotton treated with methylolmelamine. This study has been continued with fabric treated with a number of other finishing agents. Along with the beneficial effects, there is some loss of wrinkle recovery angle. This loss can be kept to a minimum with properly selected conditions for mercerization of the treated fabric. The gain in strength and abrasion resistance appears to outweigh the loss in wrinkle resistance. Well-cured samples can be mercerized to effect

the improvements and still have acceptable wrinkle recovery angles. Wrinkle resistance cottons investigated were fabrics treated with both nitrogenous and with nonnitrogenous finish agents. The former group included dimethylol cyclic ethyleneurea, methylated methylolmelamine and a methylolated triazone derivative. Formaldehyde, glutaraldehyde, pentaerythritol acetal, and an epoxy finishing agent were among those of the nonnitrogenous group. Variables in the conditions of mercerization and the results obtained are discussed. The effect of fabric construction on changes in tearing strength caused by mercerization of cloth treated with dimethylol cyclic ethyleneurea is also considered.

1659. PHYSIOLOGICAL AND BIOLOGICAL PROPERTIES OF APO AND APS, COTTON FLAME RETARDANTS. A LITERATURE REVIEW WITH BIBLIOGRAPHY
Drake, George L., Jr.; Kopacz, B. M.; and Perkerson, Fred S.
U. S. Dept. Agr. ARS 72-14, 12 pp. Processed. 1958

The publication lists 68 references relating to the physiological and biological properties of APO and APS, cotton flame retardants. The work also includes a brief review of the findings to date. In spite of the fairly large amount of work so far reported, not all of the physiological and biological properties of APO and APS are known as yet. Therefore, it is necessary to exercise normal care and caution in handling these new chemicals.

1650. EFFECTS OF COMMERCIAL LAUNDERING ON CEU-TREATED COTTON FABRICS
Mazzeno, Laurence W., Jr.; Kullman, Russell M. H.;
Reinhardt, Robert M.; and Reid, J. David
Am. Dyestuff Reptr. 47: 609-13. 1958

An untreated control, cloth finished with CEU, and with a CEU-acrylate-silicone formulation was subjected to commercial laundering at four locations to determine the effect of this type of laundering on resin treated cotton fabrics. Soiling of the sample treated with the CEU-acrylate-silicone resin was observed as a result of laundering with dirty clothing. This appears to be due to a tendency of the thermoplastic additives of this finish to attract and scavenge dirt from the laundry water. Apparently, the chlorine bleach used by these laundries did not harm the finish. The durability of the physical properties--breaking strength, tearing strength, flex abrasion, wrinkle recovery, and nitrogen content--was very good to laundering in which the souring step was well controlled. With oversouring, resin was removed by hydrolysis, resulting in decreased wrinkle recovery and an increase in most of the other properties. Investigation of the conditions of souring indicated the rate of hydrolysis to be greatly increased by elevated temperatures and by pH below 4.

1593. WET-SOILING STUDIES ON RESIN-TREATED COTTON FABRICS
Mazzeno, Laurence W., Jr.; Kullman, Russell M. H.; Reinhardt, Robert M.; Moore, Harry B.; and Reid, J. David
Am. Dyestuff Reptr. 47: P299-P302. 1958

Cotton cloth treated with a CEU-acrylate-silicone finish has been found to be subject to wet soiling, especially under conditions such as are encountered in commercial laundering. Once soiled, the CEU-acrylate-silicone finished cotton cannot be cleaned--the soiling is irreversible. CMC will decrease the amount of soiling slightly, but not sufficiently for good reconditioning. Dry-cleaning is likewise ineffective. The chief causes of the soiling are the soft, tacky films of acrylate and silicone which are deposited on the surface of the fibers. The CEU resin does not contribute to such wet soiling. In fact, cotton fabrics treated with commercial methylol resins, without additives have been found to be more resistant to wet soiling than untreated control fabrics. A simple laboratory test for wet soiling has been adopted. Cloth swatches are soiled in a Launder-Ometer with a soap solution containing a small amount of carbon under controlled conditions. A quantitative estimation of the extent of soiling is obtained, using a reflectometer. In this test, the variables--time, temperature, and concentration of carbon--affect the degree of soiling. Synthetic detergent, in place of soap, has little effect on soiling.

1666. WASH-AND-WEAR: PROGRESS AND PROBLEMS
Reid, J. David; Reinhardt, Robert M.; Kopacz, B. M.; and Fiori, L. A.
Textile Industries 122 (11): 90-98. 1958

A brief outline of the progress that has been made in the development of wash-and-wear cottons is presented. Specifically, the existing problems are reviewed with the purpose of pointing to the contributions required for all segments of the industry to produce a truly wash-and-wear product. The contributions of yarn structures and fabric geometry as a sound basis for resin treatment are pointed out. Resin treatment involves such considerations as the effects of desizing, bleaching, washing, mercerizing and dyeing. Proper selection of resin, catalyst and suitable curing and aftertreatments are also considered. Final contributions of garment construction are most important to produce the true cotton wash-and-wear garment. Through research and an understanding of all the problems the cotton textile industry is fast approaching the production of a true wash-and-wear all-cotton garment.

1577. NEW FINISHING TECHNIQUES FOR WASH-AND-WEAR COTTONS
Reid, J. David; and Reinhardt, Robert M.
Modern Textiles 39, (3): 61-68. 1958

The development of crease-resistant fabrics, their present status, and future outlook, are reviewed briefly. The resins and catalysts

now in use are discussed followed by an analysis of the mechanism of the crease-proofing process, and a discussion of the application of resins to cotton yard goods. Some of the advantages and disadvantages of cotton wash-and-wear garments are described, such as the difficulty of making smooth seams and sharp, durable creases in the finished garment, and methods to meet or avoid these problems, now under investigation. Recuring, and treatment of the finished garment are the most promising now being tried. Great advances have been made in the production of true wash-and-wear cotton garments, and the authors feel that the day of the ideal method is approaching.

1561. THE APPLICATION OF RESIN FINISHES TO COTTON GARMENTS USING DRY-CLEANING PLANT EQUIPMENT

Graham, Robert T.; Loibl, Fred; and Wiebush, Joseph R.
Contractor: National Institute of Drycleaning, Silver Spring, Md.
Textile Research J. 28: 252-56. 1958

Preliminary results are given from a study of the application of a resin finish to cotton garments using drycleaning plant equipment. Cotton fabric swatches of various constructions, including print cloth, denim, sateen, cord, broadcloth, gingham, corduroy, and suiting materials were used. The objective of the work is to impart wrinkle resistance and desirable permanent creases to cotton garments. A tentative procedure for the resin treatment has been devised which has proven effective on various weaves and weights of cotton fabrics. The cost of application of the resin finish to cotton cord suits in a drycleaning plant has been estimated.

1565. DURABLE CREASING OF WRINKLE RESISTANT COTTON

Reid, J. David; Reinhardt, Robert M.; and Kullman, Russell M. H.
Textile Research J. 28: 242-51. 1958

Research is underway at the Southern Regional Research Laboratory to improve cotton by resin treatment to strengthen its competitive position with respect to other fibers. In preliminary stages of the work, garments were finished using improved formulations and improved methods of curing which yielded garments which are truly wash-and-wear in that they may be machine laundered, dried and worn without ironing. Attention is now being concentrated on attempting to perfect a finish aimed particularly at men's "no muss" summer weight dress suit and slacks. Both fundamental and practical studies have been conducted on a "recuring" process, in which cotton goods, already treated with resin and cured, are made into garments and aftercured by careful introduction of catalyst. The creases, pleats, cuffs, and so forth are then set by heat curing. Creases so introduced are not as durable to laundering as those which can be set in the fabric on an initial

cure, but they compare favorably with those found in worsted wool garments. Two possible mechanisms for the recuring process are suggested and evidence in favor of each hypothesis is given. Results on the recuring of both laboratory and commercially resin-treated cotton fabrics are reported.

1489. AFTERMERCERIZATION OF METHYLOLMELAMINE RESIN-TREATED COTTON FABRICS

Mazzeno, Laurence W., Jr.; Reinhardt, Robert M.; Markezich, Anthony R.; and Reid, J. David
Am. Dyestuff Reptr. 46: 719-24. 1957

Aftermercerization of methylolmelamine-treated 80 x 80 print cloth improves the tearing strength of the fabric as much as 20%, and abrasion resistance up to 100%. Wrinkle-recovery angle is only slightly diminished by the treatment. Optimum conditions for this process are: resin content, 6-10%; alkali concentration, 20%; time for mercerization, 20 minutes; and temperature of mercerization, 25°C. Durability of the improvements imparted by the alkaline treatments has been shown by laundering through six standard cycles by the AATCC Method No. 14-53 modified to use 160°F. as laundering temperature. A comparison of premercerized and aftermercerized, resin-treated fabrics shows that more benefit in strength properties is achieved with the aftermercerization process. Data have been presented which qualitatively explain the action of the alkali on the resin-treated fabric.

1435. THE SURFACE OF COTTON FIBERS. PART II: MODIFIED FIBERS

Tripp, V. W.; Moore, A. T.; and Rollins, M. L.
Textile Research J. 27: 427-36, 1957

Microscopical studies of surface replicas of chemically finished or modified cotton fibers have shown that the characteristic surfaces of native cotton is often altered by various commercial and experimental treatments. While removal of the wax from the fiber surface has little effect, scouring usually uncovers the fine cellulose fibrils of the primary or even secondary walls. Mercerization does not eliminate the rugosities of the native fiber surface. Additive finishes, including starch, carboxymethyl cellulose, colloidal silica, and acrylic polymer resins change the original fiber topography to an extent dependent on the amount applied. Chemical modifications which increase the fiber cross-sectional area make the fiber surface smoother, but extensive swelling of the cellulose derivative during its preparation appears to create roughness in some modifications. Crease-resistant finishes do not alter the fiber topography significantly.

1426. STUDIES ON PERMANENT CREASING OF COTTON GARMENTS
Reid, J. David; Mazzeno, Laurence W., Jr.; Reinhardt, Robert M.; and Markezich, Anthony R.
Textile Research J. 27: 252-59. 1957

Investigations of the application of resin formulations to cotton garments for the purpose of imparting permanent creases have two phases; application to finish garments and to yard goods, with resin curing being accomplished on the finished garment in both cases. Methods of application, procedures, and results are given for application of a dimethylol cyclic ethylene urea (CEU) formulation to swatches of fabric and to garments. A number of variables such as storage, possibilities of stripping, need for precautions in drying and curing, durability to laundering, effect of sizing, effect of catalysts, and results using related resins in varying the formulation are discussed. Ultimate objectives include cooperative work with other research agencies to utilize standard drycleaning equipment for commercial application finishes suitable for white goods in imparting permanent creases to cotton garments.

1422. NOTE ON THE DETERMINATION OF THE INSOLUBILITY OF CHEMICALLY MODIFIED COTTON IN CUPRAMMONIUM HYDROXIDE SOLUTION
Schreiber, Walter T.
Textile Research J. 27: 324-26. 1957

A method is described for determining quantitatively that portion of a chemically modified cotton which is insoluble in cuprammonium hydroxide solution. The samples were treated with cuprammonium hydroxide, then with ammonium hydroxide, followed in each instance by centrifuging and decanting. A filter aid was used to assist in bringing about separation of the insoluble material, and glucose was added to inhibit decomposition.

1141. INSOLUBILITY IN CUPRAMMONIUM HYDROXIDE AS A MEANS OF DETECTING CROSSLINKING IN CHEMICALLY MODIFIED COTTON
Reeves, W. A.; Drake, G. L., Jr.; McMillan, O. J., Jr.; and Guthrie, J. D.
Textile Research J. 25: 41-46. 1955

Native cellulose dissolves in cuprammonium hydroxide solution, but, when it is crosslinked, it becomes a space polymer and is insoluble in this reagent. A method of detecting alkali-stable crosslinks in cotton is presented; the sample to be tested is shaken with cuprammonium hydroxide solution for 17 hours. If it dissolves completely, cellulose chains are not crosslinked, but if it is partially insoluble, crosslinking is indicated. Cellulose was reacted with compounds which contained one or more groups that are reactive toward cellulose. In all cases the cellulosic derivatives made from cellulose and compounds that could crosslink it were partially

insoluble in cuprammonium hydroxide. The compounds that were not capable of crosslinking in cellulose produced derivatives soluble in cuprammonium hydroxide.

797. CREASE-RESISTANT CLOTH FROM PARTIALLY CARBOXYMETHYLATED COTTON
Daul, G. C.; Reinhardt, R. M.; and Reid, J. D.
Textile Research J. 22: 792-97. 1952

When resin treatments are applied to the acid form of partially carboxymethylated cotton cloth, to produce crease-resistance, the carboxyl group supplies a built-in catalyst, making the addition of catalyst unnecessary, in this way avoiding the pre-polymerization of resin baths which contain added catalysts. The high swellability of the modified cotton allows easier penetration of the resin-formers and larger pickup of resin, with the production of cloth of equivalent crease-resistance, and superior in most other physical properties to unmodified, resin-treated cloth. The process of carboxymethylation (impregnation of cotton cloth with a weak solution of monochloroacetic acid followed by treatment with strong sodium hydroxide) is commercially feasible and can be carried out on ordinary textile equipment.

532. REACTION OF FORMALDEHYDE WITH COTTON
Goldthwait, Charles F.
Textile Research J. 21: 55-62. 1951.

Partially methylenated cottons were prepared by the reaction of cotton with formaldehyde under acid conditions in nonaqueous (mainly acetone) solutions. The modified cotton, although it did not differ from ordinary cotton in appearance and general textile character, displayed new properties even at relatively low formaldehyde contents (from 0.5 percent to 1.5 percent) which may prove to be of practical importance. Dye-resisting yarns of possible technological interest can be produced, apparently, mainly owing to crossbonding in the cellulose. The modified cotton has a decreased swelling capacity in water and other swelling agents. Products of sufficient formaldehyde contents have good resistance to biological rotting. Direct cotton dyes are made faster by the treatment.

39. DETERMINATION OF COMBINED FORMALDEHYDE IN ORGANIC COMPOUNDS AND IN CELLULOSE FORMALS
Hoffpauir, C. L.; Buckaloo, G. W.; and Guthrie, J. D.
Ind. Eng. Chem. 15 Anal. Ed. 605-06. 1943

Standard methods for the determination of formaldehyde usually require distillation with acid in order to isolate the formaldehyde. In the method presented, distillation is omitted and the formaldehyde is determined in the hydrolyzate by a procedure adapted to a photoelectric colorimeter in which a magenta color is developed by use of Schiff's reagent. Correct results may be obtained even

in the presence of considerable amounts of other aldehydes. The procedure is applicable to organic formals and because of its sensitivity can be used on samples containing very small amounts of formaldehyde.

789.1 ACTION OF CYANURIC CHLORIDE ON COTTON CELLULOSE
Warren, Jane; Reid, J. David; and Hamalainen, Carl
Textile Research J. 22: 584-90. 1952

The reaction of cyanuric chloride with alkali-treated cotton cellulose has been examined to determine the effect of reaction conditions on degree of substitution and on retention of chlorine for further modification. Type of alkali pretreatment and choice of organic solvent were found to influence the reaction. The highest substitution obtained was 1 triazine ring per 1.9 anhydroglucose residues, based on nitrogen content. Although the highest chlorine content was 1 chlorine atom per 3.4 anhydroglucose residues, this residual chlorine was stable to heating at 101°C. and to several months of storage, and was capable of reacting with ethylenediamine and hexamethylenediamine. Strength loss during treatment was low and in no case exceeded 26% of the original. A change in dyeing properties was found to be related to degree of triazine substitution in some cases and to type of pretreatment in others. The treated yarn had some resistance to microbiological attack.

WASH-WEAR AS REPORTED AT SOME CONFERENCES

WASH-WEAR COTTONS: REPORT OF PROGRESS AND CURRENT STATUS - PROCEEDINGS OF THE CONFERENCE OF COLLABORATORS FROM SOUTHERN AGRICULTURAL EXPERIMENT STATIONS

March 28-30, 1960

The current status of wash-wear research at the Southern Utilization Research and Development Division is presented in eleven of the 17 papers on wash-wear presented at the two-day conference. Papers on various phases of the wash-wear situation were also presented by representatives of the National Cotton Council of America, industry, and state and federal workers. Titles in the Proceedings are: Economic Values and Trends in Wash-Wear; Current Problems in Wash-and-Wear; Multipurpose Wash-Wear Cotton Fabrics; Studies in Chemical Finishing; New Products and Processes for Wash-and-Wear; Effect of Fabric Construction on Wash-Wear Properties of Wrinkle Resistant Cotton Fabrics; Wash-and-Wear Fabric Evaluation Using the Electronic Smoothness Evaluators; Improvements in Wash-and-Wear Through Garment Evaluation; Wash-and-Wear Cotton from the Retailers' Point of View; Cleaning and Wrinkling Effects of Low Temperature Wash Solutions; Further Progress in Wash-Wear; Research of the Western Division on Wool Wash-Wear; The Reaction of Cotton with Formaldehyde in Solution; New Finishes for Wash-Wear Cotton; Studies on Cotton Finished with Epoxy Compounds; Testing of Wash-and-Wear Fabrics; Microscopical Examination of Resin Treated Cotton; Fiber Properties Related to Wash-Wear Treatments; Brief Survey of the Research Program of the Southern Division; Research on Oilseeds and Industrial Crops; SU Research on Food Crops.

ADVANCES IN RESEARCH ON WASH-WEAR GARMENTS - NEWER ANALYTICAL TECHNIQUES AND INSTRUMENTATION IN PRODUCTION AND UTILIZATION RESEARCH - PROCEEDINGS OF THE CONFERENCE OF COLLABORATORS FROM SOUTHERN AGRICULTURAL EXPERIMENT STATIONS

April 13-15, 1959

Presented during the section of the program devoted to wash-wear were reports by members of the staff of the Southern Utilization Research and Development Division discussing new research developments in this field. The program also includes several papers giving the industry viewpoint. The section of the program reviews research by the Southern Division in other areas and descriptions of analytical procedures and techniques. Titles of papers are: A Birdseye View of the Present Status of Wash-and-Wear Cottons; Industrial production of Wash-Wear Materials; Problems and Opportunities in Developing Improved Wash-and-Wear Cotton Products; Rot and Wrinkle Resistance Using Acid Colloid of Melamine; Durable Crease Using Strong Caustic Reagent; Wrinkle Resistance and Flame Resistance Using APO; New Wash-and-Wear Finishing Agents of Industry and SU; Chemical Modification of Wet Wrinkle Resistance; The Need for Research on the Influence of Fabric Structure and Related

Factors on the Quality of Wash-Wear Garments; Laboratory Methods for Evaluating Wash-and-Wear Properties; Physical Tests on Fibers, Yarns, and Fabrics; Application of X-Rays in Analytical and Structural Investigation; Infrared Absorption Spectroscopy - A Valuable Tool of Chemical Analysis; Application of Glass Paper Chromatography to the Isolation, Identification, and Quantitative Estimation of Natural Products; Chromatography in the Study of Sugarcane Juice; The Ionization Detection System Gas Chromatography; Atomic Energy Applications in Agricultural Research; Sampling for Analysis - New Approaches; The Present and Probable Impact of Wash-Wear Developments on Cotton Agriculture; Economic Implications and Market Potentials of Wash-and-Wear Textiles; The Impact of New and Improved Analytical Techniques on Agricultural Research.

FLAMEPROOFING OF COTTON WITH THPC RESINS
Reeves, W. A.; and Guthrie, J. D.
Proc. 2nd Cotton Chem. Finishing Conf., 1953, 65-70.

A rather unusual chemical compound has been found which can be applied in combination with other chemicals to cotton fabric to make it permanently flame-resistant. This crystalline compound is tetrakis (hydroxymethyl) phosphonium chloride, $(HOCH_2)_4PCl$, abbreviated "THPC."

The flameproofing treatment can be applied on standard chemical finishing equipment. An aqueous solution of THPC, trimethylolmelamine, urea, and triethanolamine is applied to fabric with a padder with a tight nip roll pressure to keep the wet pick-up at a minimum. The fabric is then dried at a relatively low temperature, cured at an elevated temperature, and washed by any of the usual washing procedures. A softener should be applied to the treated fabric.

Eight ounces twill containing about 16 percent of the THPC-resin will pass the standard vertical flame test. The resin is permanent to laundering. In addition to being flameproof, treated fabric is also glowproof. Considerable wrinkle resistance and rot or mildew resistance are imparted by the flameproofing treatment.

CARBOXYMETHYLATION - CHEMICAL BONDING WITH RESINS - TREATMENT WITH OCTADECYL ISOCYANATE - OTHERS
Reid, J. David
Proc., The Cotton Chem. Finishing Conf., 1952, 32-33.

The chemical modification of cotton cellulose gives what is essentially a new fiber, changing the chemical composition of the cellulose but retaining the fibrous character of the cotton. A number of methods of obtaining products with changed properties have been investigated. Three widely differing methods of chemical modification have been selected as illustrative examples, including carboxymethylation, chemical bonding with a resin-forming chemical, beta propiolactone, and topochemical reaction with a very small amount of octadecyl isocyanate. Carboxymethylation gives

a superior creaseproofness without the stiffness usually associated with a high resin content. Addition of 0.2 percent of octadecyl isocyanate gives good spray resistance.

FLAMEPROOFING OF COTTON WITH PHOSPHORUS COMPOUNDS
Guthrie, John D.
The Cotton Chem. Finishing Conf., 1952, 31.

A promising flame retardant application has been found, based on impregnation of cotton with a polymer containing phosphorus has been found. This polymer is based on tetrakis (hydroxymethyl) phosphonium chloride (THPC). When it is mixed with methylolmelamine in aqueous solution, applied to cotton and cured, excellent flame resistance of the permanent type if obtained. Crease resistance is also imparted to the fabric, but the loss in tear strength may be a disadvantage in some applications. Work is in progress to minimize this decrease in tear strength.

THE CONCEPT OF CHEMICAL FINISHING
Fisher, C. H.
Proc. The Cotton Chem. Finishing Conf., 1952, 6-11.

Theories, methods, and mechanism of chemical modification of cotton are discussed, and an analogy drawn between the chemical modification of cotton and the production of synthetic fibers. Various advantages of chemically modified cottons are discussed, followed by an exploration of the many possible treating agents and products. Crosslinking and polymerization of the treating agents are discussed. Among the possible chemical modifications are treatments in which substituent groups might be attached to the cellulose chain, such as bulky side groups, graft polymers, swelling, decrystallization, and crosslinking. The speaker expressed the belief that cotton could be so modified that cotton fabrics would not need ironing, or at least would require much less ironing than now.

IMPROVEMENT OF CREASE RESISTANCE AND IMPARTING CREASE RESISTANCE TO COTTON TEXTILES
Cotton Research Clinic, 1950, 19, 50-51.

James Dean, who discussed the improvement of crease resistance, gave a general review of past and current crease resistance work at SRRL. He said that melamine formaldehyde treatments were not as efficient as urea formaldehyde treatments. Variations in yarn twist, ends and picks, and other fabric construction factors had been correlated with resistance to creasing after processing with melamine formaldehyde resins. Research or crease resistance at the Southern Laboratory was discussed in greater detail during the open forum discussion on cotton quality research, at which time the desirability of improved wrinkle resistant finishes at moderate cost was emphasized.

Patents*

1970. FLAME-RESISTANT CELLULOSIC TEXTILES AND PROCESS FOR REACTING CELLULOSE ETHER TEXTILES WITH AZIRIDINYL PHOSPHINE OXIDE OR SULFIDE

U. S. Pat. No. 2,933,367, April 19, 1960
Reeves, Wilson A.; Drake, George L., Jr.; and Guthrie John D.

Flame-resistant textiles produced by the reaction of aziridinyl phosphine oxide or sulfide with chemically modified cellulose.

1903. PROCESS FOR THE PRODUCTION OF CELLULOSIC TEXTILES WITH PERMANENT CREASES AND IMPROVED SOIL AND ABRASION RESISTANCE

U. S. Pat. No. 2,917,412, December 15, 1959
Reinhardt, Robert M.; Mazzeno, Laurence W., Jr.; and Reid, John D.

A process for treating textiles to produce a finish thereon which will cause the material to retain permanent creases, exhibit wrinkle-resistance, dimensional stability, and resistance to soiling. The textile is treated with a water emulsion containing dimethylol cyclic ethylene urea, emulsified polyethylene, a latent acid catalyst, and an emulsifier.

1897. FLAME- AND CREASE-RESISTANT TEXTILES FROM AZIRIDINYL CARBOXYALKYLCELLULOSE

U. S. Pat. No. 2,906,592, September 29, 1959
Reeves, Wilson A.; Guthrie, John D.; and Drake, George L., Jr.

Flame-resistant, crease-resistant textiles prepared by treatment with the reaction product of compounds containing two or more aziridinyl groups and carboxyalkylcellulose.

1803. COMPOSITION COMPRISING NITRILO METHYLOL-PHOSPHORUS-POLYMER AND ORGANIC TEXTILES FLAME-PROOFED THEREWITH

U. S. Pat. No. 2,892,803, June 30, 1959
Reeves, Wilson A.; and Guthrie, John D.

Polymers produced by condensation or esterification reactions between methylol phosphorus compounds (THPC and/or THPO) and nitrilo compounds (compounds with a molecular weight below 800 and containing at least one nitrogen atom having attached

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*Some of the patents listed do not mention wrinkle resistance, but are included because the developments contribute to subsequent work in this area, for example, the aziridinyl compounds.

thereto hydrogen atoms of CH_2OH groups). Textile fabrics treated with solutions of the prepolymer ingredients exhibit no after-flame properties (i.e. do not continue to burn following removal of the igniting flame).

1791. FLAME RESISTANT ORGANIC TEXTILES AND METHOD OF PRODUCTION
U. S. Pat. No. 2,870,042, January 20, 1959
Chance, Leon H.; Drake, George L., Jr.; and Reeves, Wilson A.

The process for flameproofing textile materials. The flameproofing process employs mixed polymers produced from 1-aziridinyl phosphine oxide or sulfide and polyhydric alcohols.

1707. BROMINE CONTAINING NITRILO METHYLOL-PHOSPHORUS POLYMERS
U. S. Pat. No. 2,861,901, November 1958
Reeves, Wilson A.; Hamalainen, Carl; and Guthrie, John D.

This invention relates to crosslinked liquid or solid polymers which contain phosphorus, nitrogen, and bromine in the form of stable chemical components. The polymers have utility for reducing the flammability of textiles.

1706. FLAME RESISTANT ORGANIC TEXTILES AND METHOD OF PRODUCTION
U. S. Pat. No. 2,859,134, November 4, 1958
Reeves, Wilson A.; Chance, Leon H.; and Drake, George L.

Flame resistant polymers and the processes for producing flame resistant textiles by use of these polymers are covered by this patent. The polymers are produced by the reaction of 1-aziridinyl phosphine oxide or sulfide with an amino compound which has one or more radicals consisting of hydrogen or CH^2OH attached to a trivalent nitrogen atom.

1258. ALKALI METAL SALT OF PROPARGYL HYDROGENSULFURIC ACID AND PROCESS OF MAKING UNSATURATED CELLULOSE ETHER TEXTILES
U. S. Pat. No. 2,727,805, December 20, 1955
Parker, Edwin D.; and Guthrie, John D.

A process is described for making partial unsaturated ethers of cotton by reacting it with sodium allyl sulfate or sodium propargyl sulfate in the presence of sodium hydroxide.

714. METHOD OF MAKING PARTIALLY CARBOXYMETHYLATED OR PHOSPHORYLATED CELLULOSIC FIBROUS MATERIALS CREASE RESISTANT AND DYE RESISTANT
U. S. Pat. No. 2,584,114, February 5, 1952
Daul, G. C.; and Reid, J. D.

The method comprises impregnating the materials with an acid-polymerizable substance, such as a melamine-formaldehyde pre-condensate, and heating to polymerize the latter, in the absence of an added external catalyst. The method produces cloths which are free of acid-induced degradation and possess excellent hand, abrasion, resistance, and other properties.

Patent Applications

PC 3546 -- "Process for the Production of Permanent Creases in Cellulosic Textiles," by C. H. Mack and W. A. Reeves

A process for producing permanent creases in cellulosic textiles either untreated or those which already contain a resin finish. The process comprises wetting the textile with a solution of strong base and setting the crease by heating with pressure.

PC 3551 -- "Wrinkle Resistant Cellulosic Textiles and Processes for Producing Same," by L. H. Chance and W. A. Reeves

A process for producing crease resistant cellulosic textiles which comprises treating the textile with either carbonylbisaziridine or thiocarbonylbisaziridine.

PC 3585 -- "Wrinkle Resistant Cellulosic Textiles and Method of Production," by G. L. Drake, L. H. Chance, W. A. Reeves, and J. D. Guthrie

A wrinkle resistant treatment for textiles produced by applying 1-aziridinylphosphine oxide or sulfide in the presence of a latent acid catalyst.

PC 3611 -- "Improved Processes for Treating Cellulosic Textiles with Acid Colloids of Methylolmelamine," by W. A. Reeves and W. N. Berard

A resin treatment for cellulosic textiles characterized by the use of colloidal solutions (unaged aqueous formic acid) of methylolmelamine.

PC 3614 -- "Wrinkle Resistance Treatment for Cellulosic Textile Fabrics," by R. M. Reinhardt, J. G. Frick, Jr., R. L. Arceneaux, and J. D. Reid

A wrinkle resistant treatment for cellulosic textile fabrics which is immune to damage from hypochlorite bleaching agents. The resin treatment employs dimethylol ethyleneurea, and a polymeric acetal formed from formaldehyde and a polyhydroxy compound.

PC 3620 -- "Wrinkle Resistance Treatment for Cellulosic Textile Materials," by J. G. Frick, Jr.

A wrinkle resistant treatment for cellulosic textile materials which consist of treating the fabric with the monomeric reaction product of cyanuric acid and formaldehyde and heat curing the treated material.

PC 3712 --"Wrinkle Resistant Cellulosic Textiles and Method of Production,"by S. L. Vail, J. G. Frick, Jr., and J. D. Reid

A method for preparing N-alkylmelamines. These materials upon reaction with formaldehyde produce water soluble methylolmelamine that have outstanding characteristics as treating agents for cellulosic textiles. The new N-alkylmelamines prepared according to the process of this invention confer increased resiliency and wrinkle resistance upon the fabrics to which they are applied. The finished fabrics are able to stand repeated laundering and the treated fabrics resist hypochlorite damage.

PC 3747 -- "Treatment of Fibrous Cellulosic Materials with Formaldehyde," By W. A. Reeves, R. M. Perkins, and L. H. Chance

An improved process for treating cotton cellulosic textiles with formaldehyde. This process is characterized by the careful control of fiber swelling and reagent concentration throughout the treatment. Wet and dry wrinkle resistance is imparted to the fabric as a result of this formaldehyde treatment.

PC 3780 -- "Process for Producing Wrinkle Resistant Cellulosic Textile Materials," by Bethlehem K. Andrews

A process for treating cellulosic textiles in order to make them resistant to creasing and wrinkling. The process of this invention involves the reaction product of formaldehyde and the ureide of mesooxalic acid.

PC 3783 -- "Dyed Cellulosic Textiles and Processes for their Production," by W. A. Reeves, G. L. Drake, Jr.; and J. D. Guthrie

A process for attaching dyes by chemical bonding to cellulosic materials. The bonding is accomplished by the reaction of aziridinyl groups with halotriazines and cellulosic hydroxyls. The fabrics treated according to the process of this invention are durable to repeated launderings, have improved dimensional stability, rot resistance, and wrinkle resistance.

PC 3798 -- "Wrinkle Resistant Cellulosic Textiles and Methods of Production," by S. L. Vail and P. J. Murphy, Jr.

Wash-wear treatment for cellulosic textile fabrics in which the treated fabrics are rendered resistant to wrinkling and exhibit improved resiliency. The treatment is resistant to laundering and attendant hypochlorite bleaching. The process involves treating the cellulosic textile with an aqueous solution containing 1,3-dimethyl-4,5-dihydroxy-2-imidazolidinone.

PC 3817 -- "Process for Making N-Substituted Aminoethylsulfonylethyl Esters of Cellulose," by Clark M. Welch and John D. Guthrie

A method for attaching organic amines to cellulose with divinylsulfone. The process of this invention offers a method of attaching water repellents, rot proofing agents, stiffening agents, flame retardants, and other textile finishing agents which are amines to cotton. Under suitable conditions durable wrinkle resistance in the wet and dry state can be imparted to fabrics.

PC 3826 -- "Process for Treating Cellulosic Thread to Eliminate Seam Pucker," by J. D. Reid, T. W. Fenner, and Julia M. Sloan

A process for treating a cellulosic thread with a resin so that the treated thread can be sewn into the garment and resin cured in place. This method of operation establishes stability and eliminates seam pucker.

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